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Electron Backscatter Diffraction (EBSD) Characterization of Uranium and Uranium Alloys

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Electron backscatter diffraction (EBSD) was used to examine the microstructures of unalloyed uranium, U-6Nb, U-10Mo, and U-0.75Ti. For unalloyed uranium, we used EBSD to examine the effects of various processes on microstructures including casting, rolling and forming, recrystallization, welding, and quasi-static and shock deformation. For U-6Nb we used EBSD to examine the microstructural evolution during shape memory loading. EBSD was used to study chemical homogenization in U-10Mo, and for U-0.75Ti, we used EBSD to study the microstructure and texture evolution during thermal cycling and deformation.

The studied uranium alloys have significant microstructural and chemical differences and each of these alloys presents unique preparation challenges. Each of the alloys is prepared by a sequence of mechanical grinding and polishing followed by electropolishing with subtle differences between the alloys. U-6Nb and U-0.75Ti both have martensitic microstructures and both require special care in order to avoid mechanical polishing artifacts. Unalloyed uranium has a tendency to rapidly oxidize when exposed to air and a two-step electropolish is employed, the first step to remove the damaged surface layer resulting from the mechanical preparation and the second step to passivate the surface. All of the alloying additions provide a level of surface passivation and different one and two step electropolishes are employed to create good EBSD surfaces.

Because of its low symmetry crystal structure, uranium exhibits complex deformation behavior including operation of multiple deformation twinning modes. EBSD was used to observe and quantify twinning contributions to deformation and to examine the fracture behavior. Figure 1 shows a cross section of two mating fracture surfaces in cast uranium showing the propensity of deformation twinning and intergranular fracture largely between dissimilarly oriented grains.

Deformation of U-6Nb in the shape memory regime occurs by the motion of twin boundaries formed during the martensitic transformation. Deformation actually results in a coarsening of the microstructure making EBSD more practical following a limited amount of strain. Figure 2 shows the microstructure resulting from 6% compression.

Casting of U-10Mo results in considerable chemical segregation as is apparent in Figure 2a. The segregation subsists through rolling and heat treatment processes as shown in Figure 2b. EBSD was used to study the effects of homogenization time and temperature on chemical heterogeneity. It was found that times and temperatures that result in a chemically homogeneous microstructure also result in a significant increase in grain size.

U-0.75Ti forms an acicular martinsite as shown in Figure 4. This microstructure prevails through cycling into the higher temperature solid uranium phases.

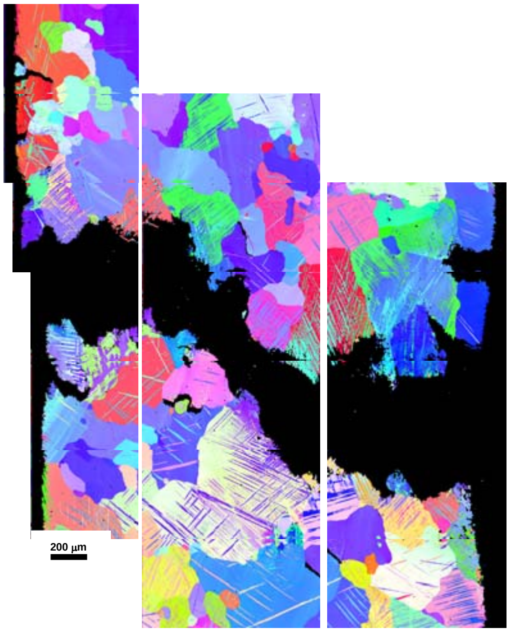

Figure 1. Cross section of mating fracture surfaces in an unalloyed uranium tensile specimen. Apparent is the prevalence of deformation twins and the intergranular fracture between grains of dissimilar orientation

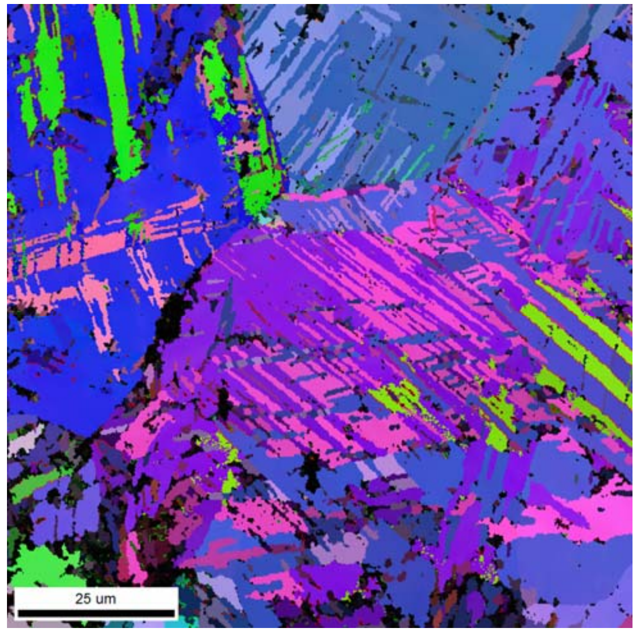

Figure 2. Microstructure of U-6Nb following 6% compressive deformation.

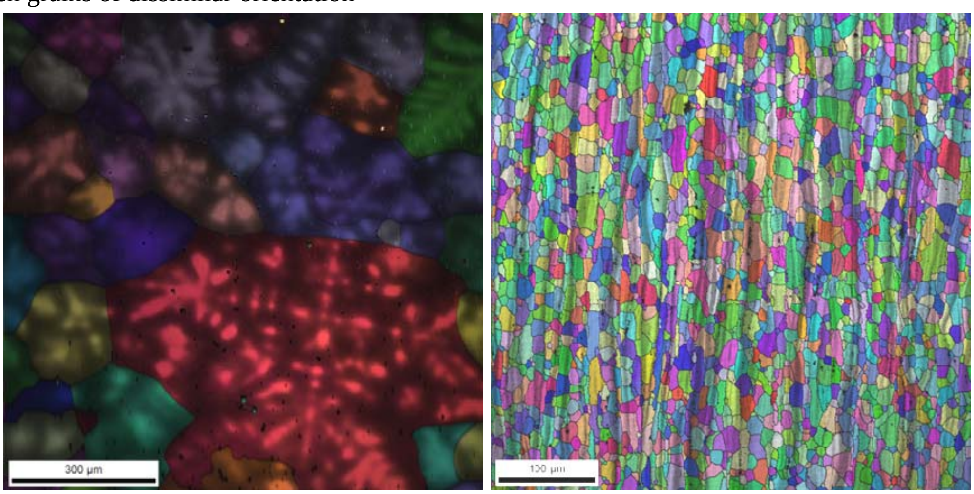

Figure 3. Microstructures of U-10Mo showing chemical segregation following a) casting and b) hot rolling and recrystallization.

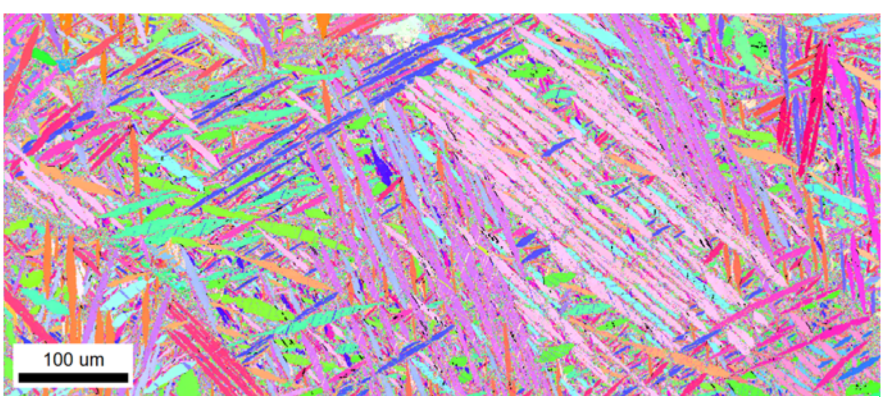

Figure 4. Acicular martensite observed in U-0.75Ti

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Materials Science & Technology Division, Materials Technology - Metallurgy (MST-6)

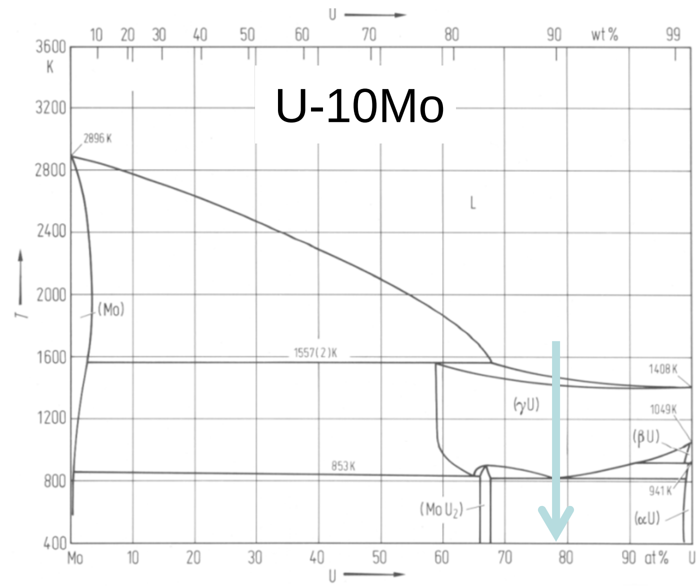
Los Alamos National Laboratory

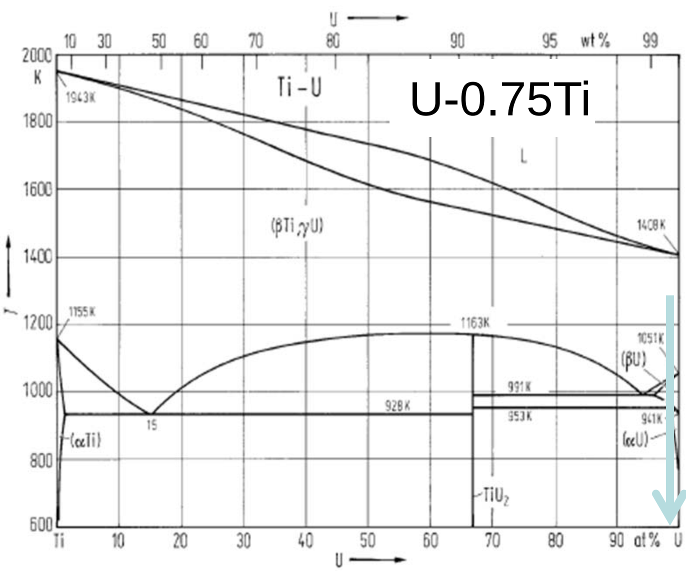
EBSD of Uranium Alloys

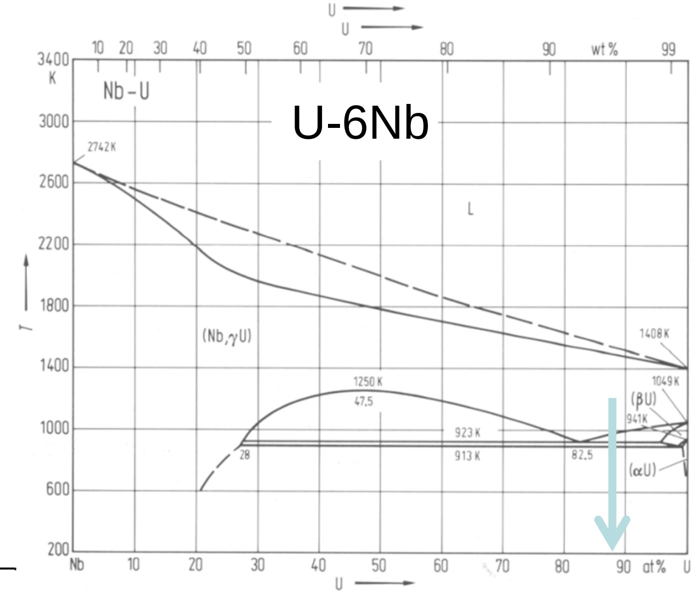
- Challenges
- Specimen preparation
- EBSD of Uranium Alloys
  - ➢ U-10Mo
  - ➢ U-6Nb
  - ➢ U-0.75Ti
  - ➢ Unalloyed Uranium

Uranium Alloy Challenges

- Specimen handling
- Oxidation
- Microstructure
  - ➢ Unalloyed Uranium
  - ➢ U-10Mo, U-6Nb, U-0.75Ti





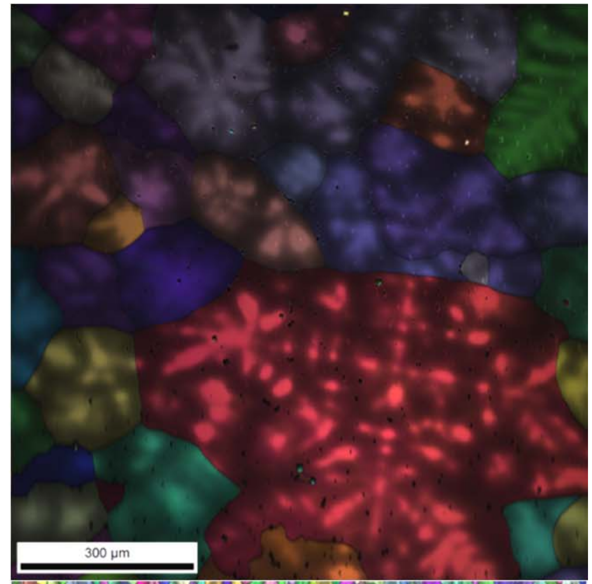


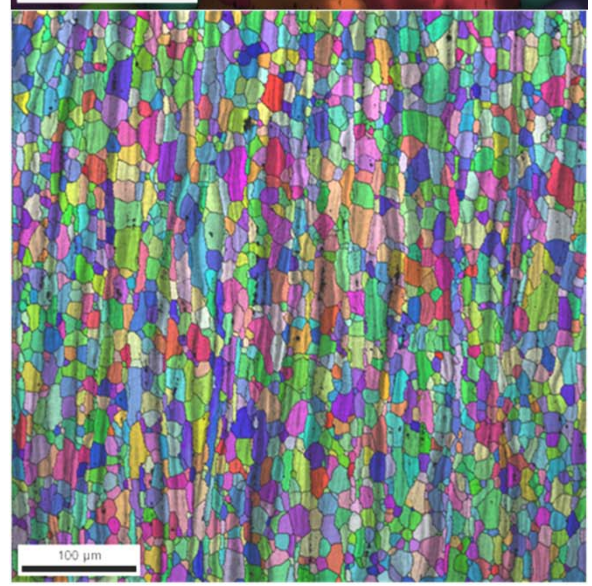
Uranium Alloy Sample Preparation

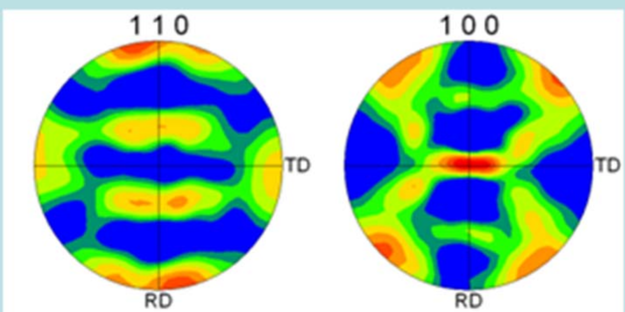
- Mechanical grinding/polishing to 1μm diamond – co-rotation for alloys at 1μm
- Electropolish A
  - 27 H_3PO_4 : 45 ethanol : 27 ethylene glycol
  - 10-20 V, 5-10 minutes, stirred
- Electropolish B
  - 5 H_3PO_4 : 95 H_2O
  - 5 V, 1-2 seconds
- Electropolish C
  - 85 H_3PO_4 : 2 H_2SO_4 : 13 H_2O :
  - 12 V, 5 seconds

U-10Mo Alloy Fuels

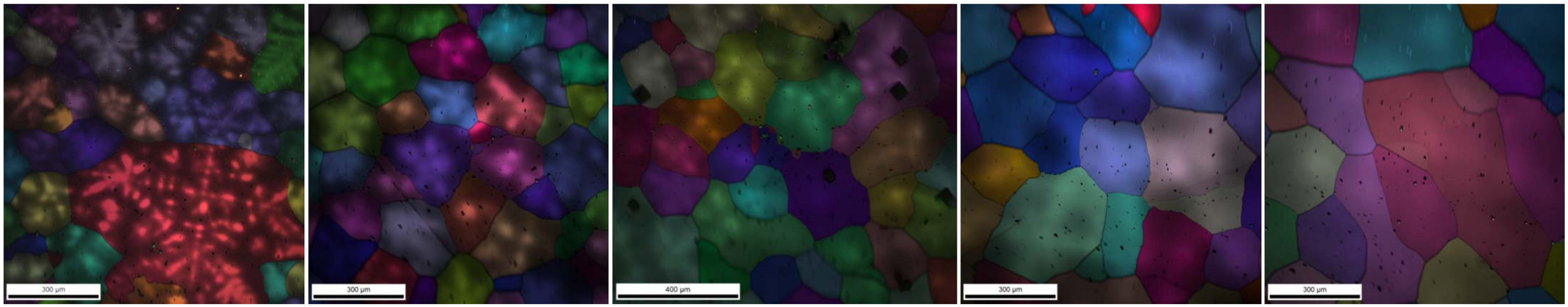
- Mo segregation in as-cast microstucture
  - ~100 µm grain size
- Segregation persists following hot rolling and annealing
- Typical BCC rolling texture



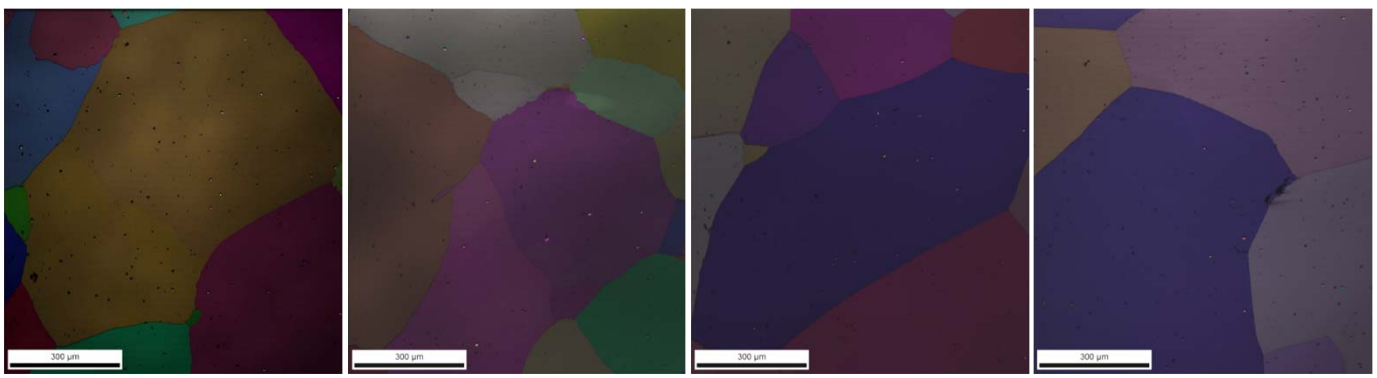




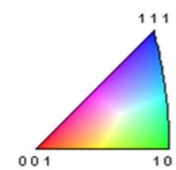
U-10Mo Casting - Homogenization



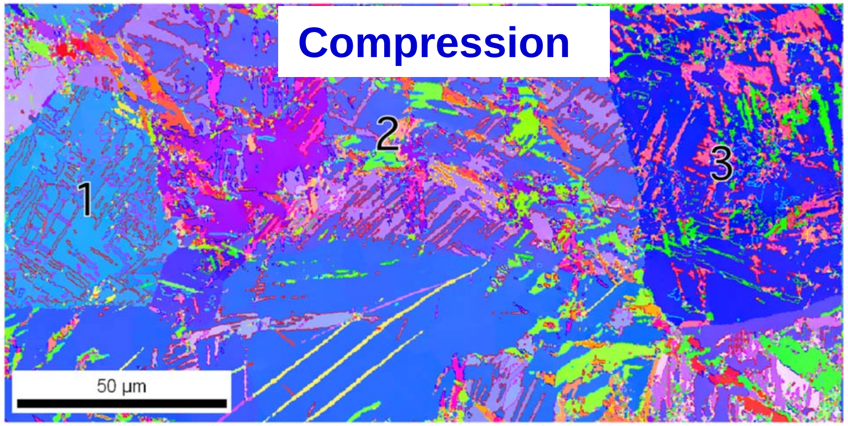
Cast | 800°C, 4 h | 800°C, 16 h | 1000°C, 1 h | 1000°C, 2 h

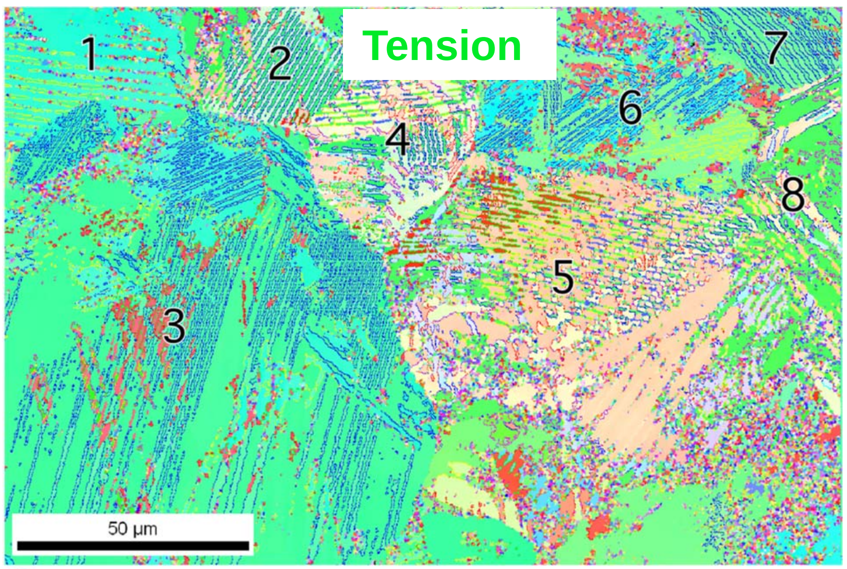


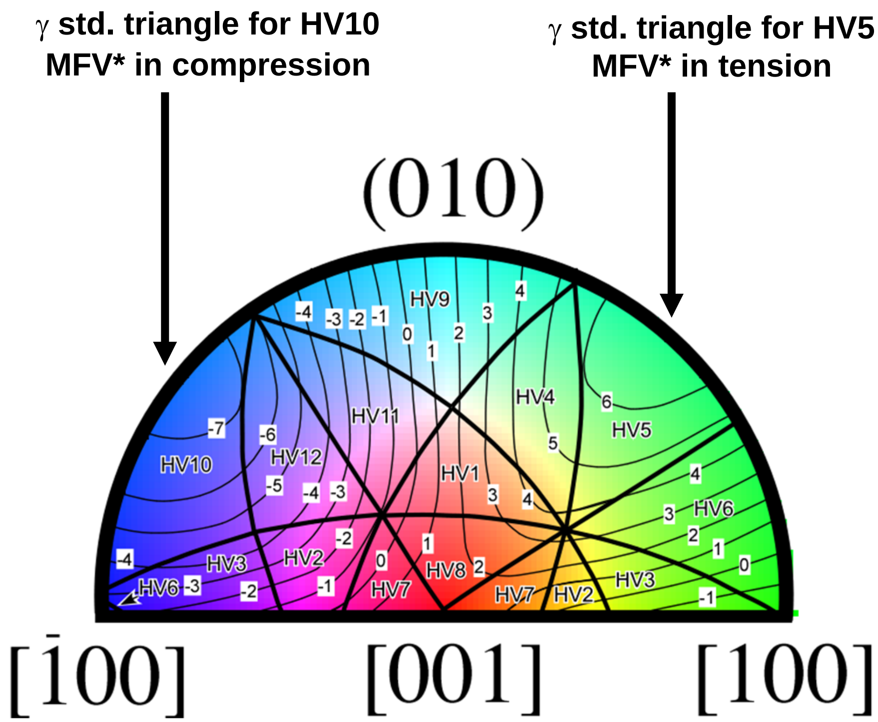
1000°C, 4 h | 1000°C, 8 h | 1000°C, 16 h | 1000°C, 32 h



U-6Nb Deformation





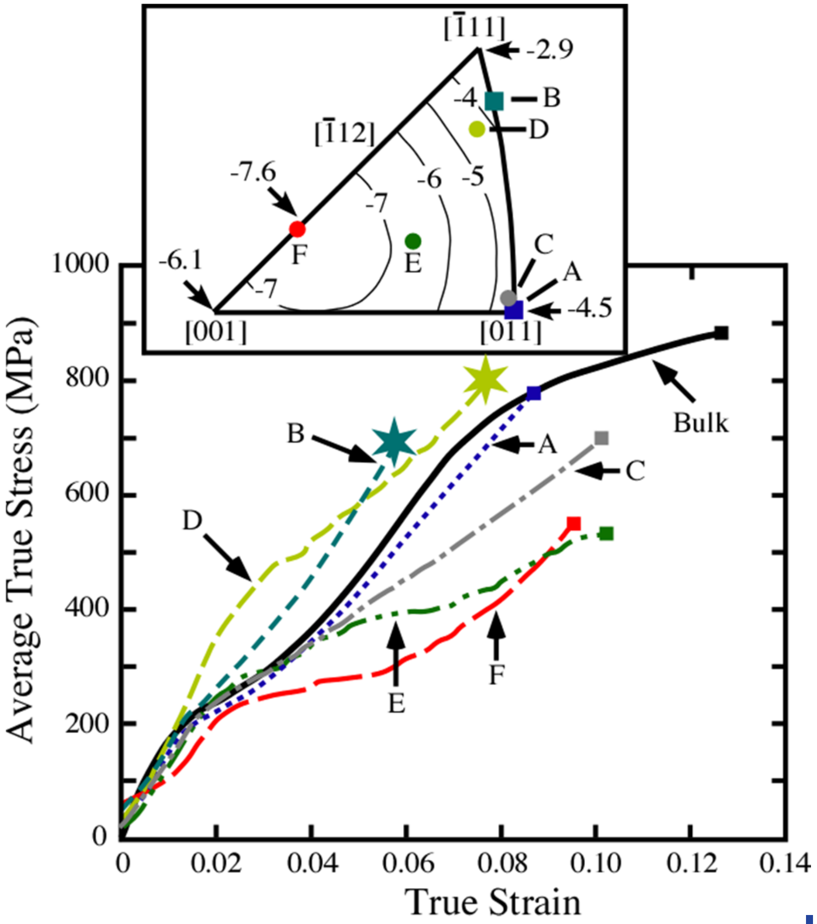


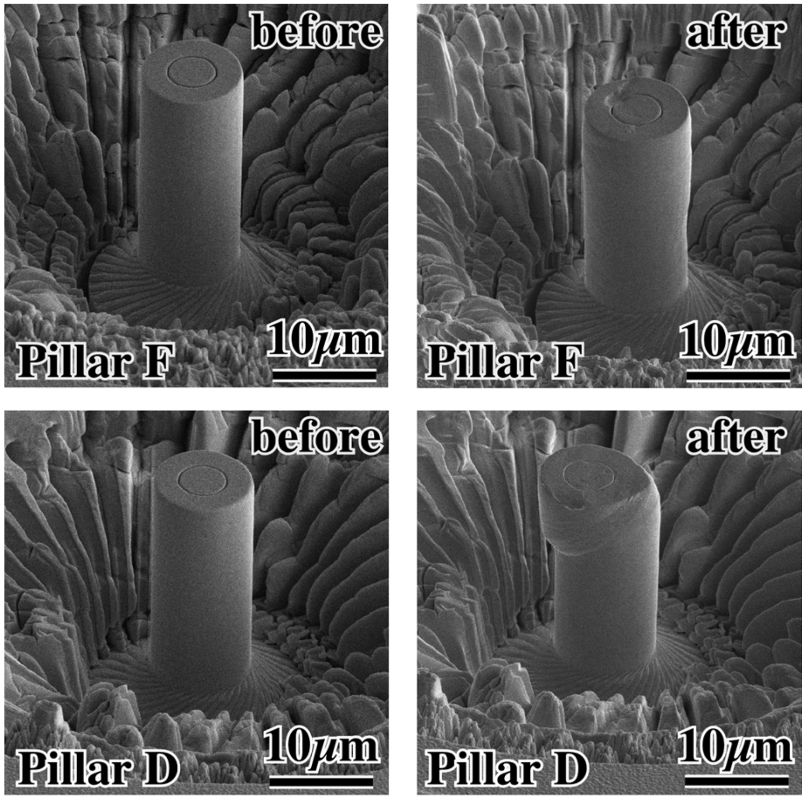
Martensite orientation color legend, position of γ standard triangles for the 12 possible martensite Hatt† variants (HVs) determined from the known orientation relationship, and calculated accommodation strain contours.

†Hatt BA. J Nucl Mater 1966; 19:133

*** Most Favored Variant**

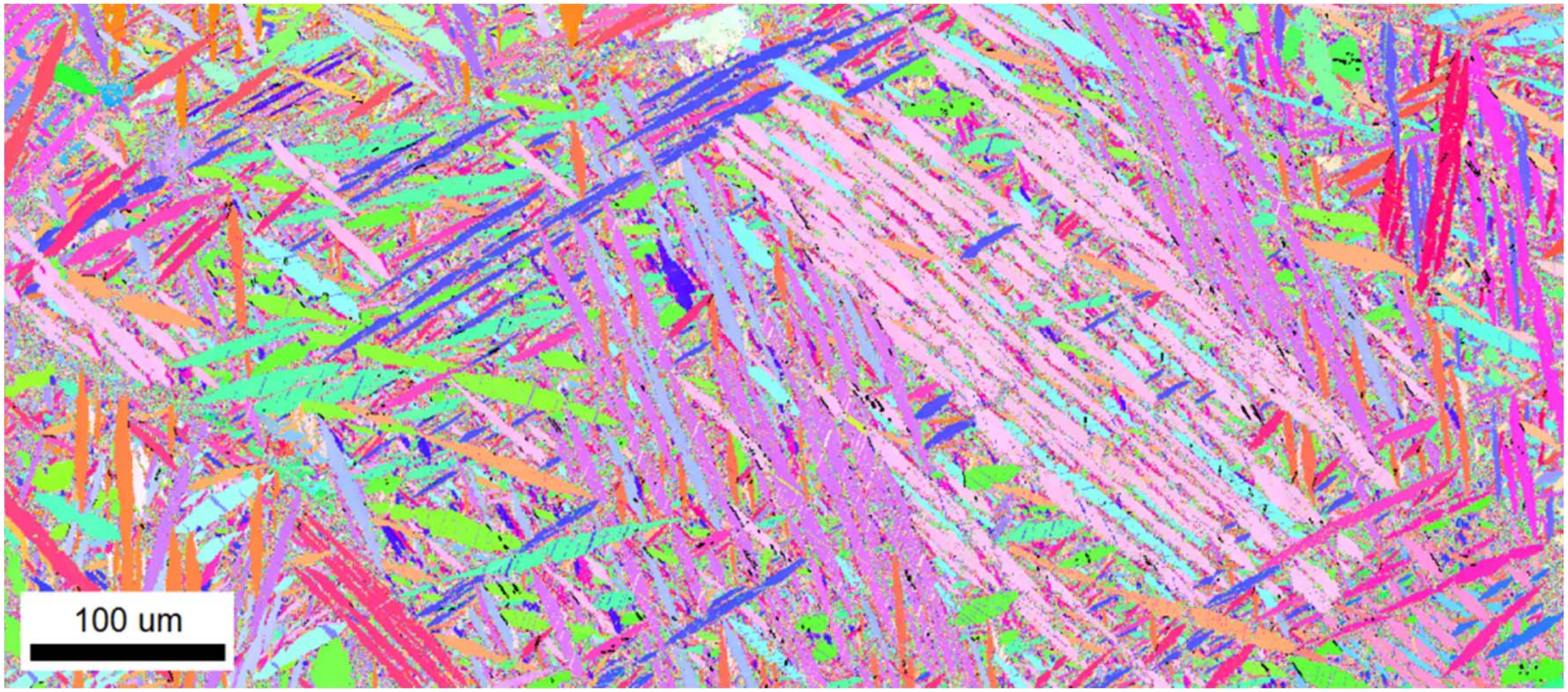
U-6Nb: Micro-Pillar Testing





Orientations with higher accommodation strains are "soft" and orientations with lower accommodation strains are "hard", in agreement with single crystal predictions

U-0.75 Ti: Texture Memory



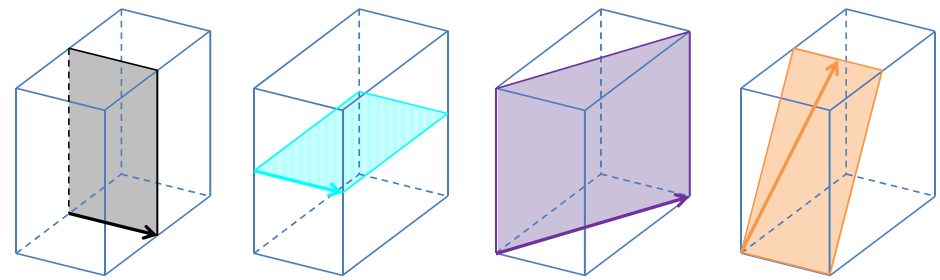
α-Uranium

Orthorhombic uranium

6-8 deformation modes

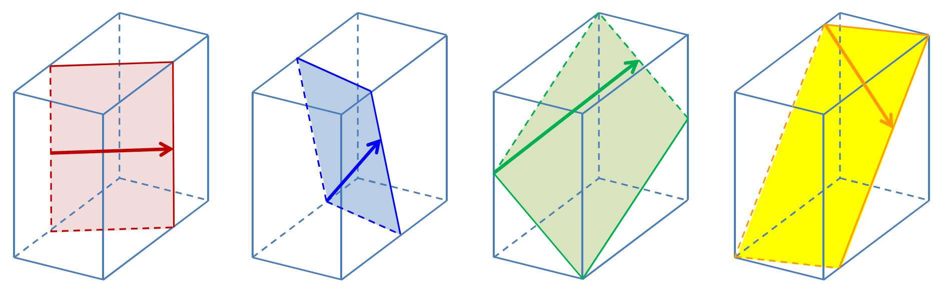
Uranium exhibits complex deformation physics

Slip Modes



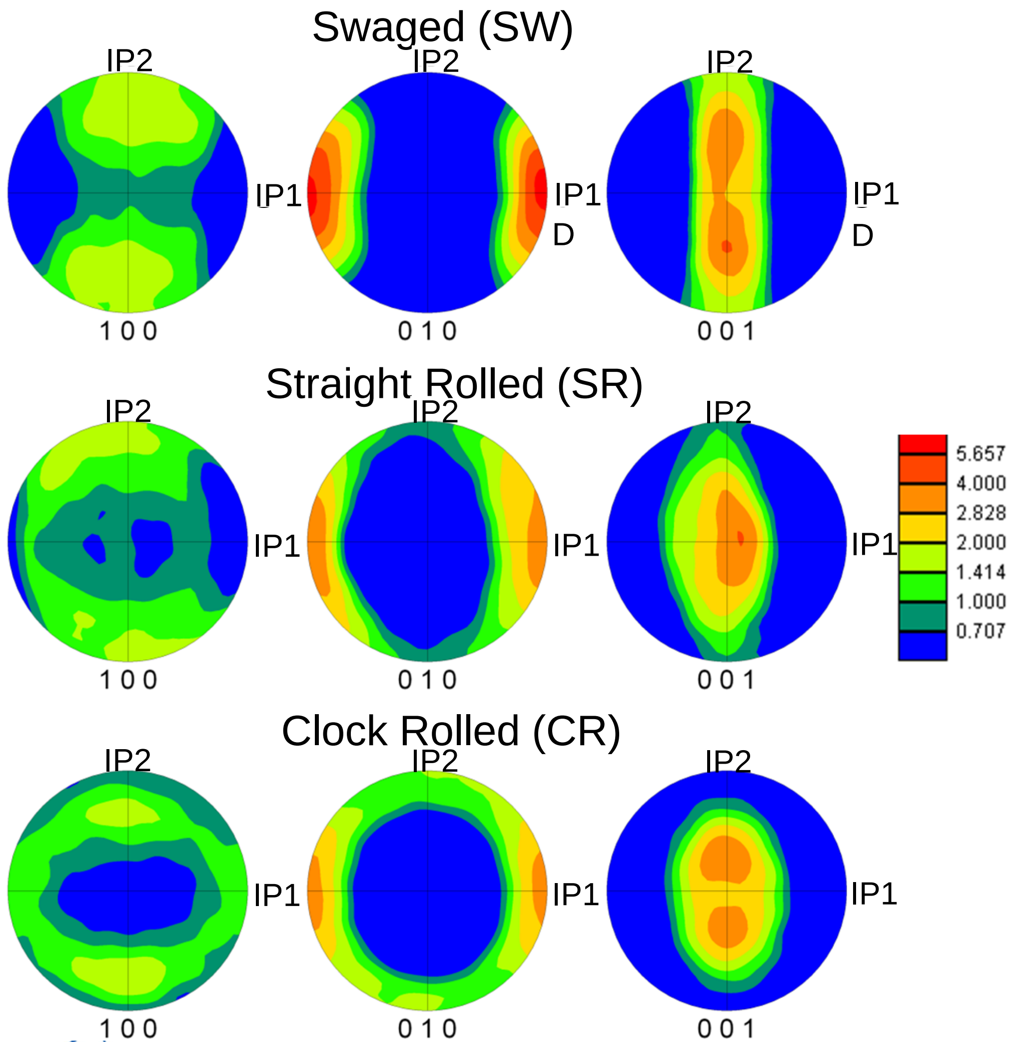
(010)[100]	(001)[100]	$\{110\}\langle 1\bar{1}0\rangle$	$\{021\}\langle 1\bar{1}2\rangle$
1 Slip System	1 Slip System	2 Slip Systems	4 Slip Systems

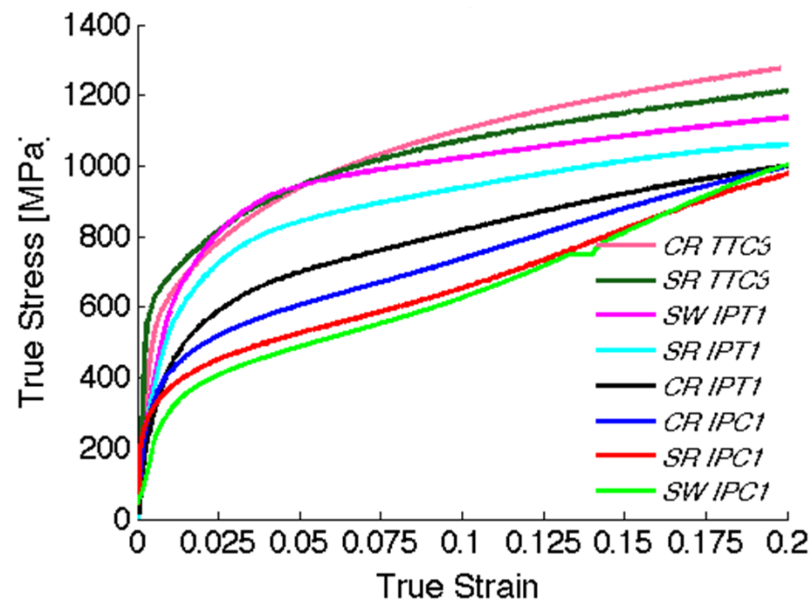
Twin Modes



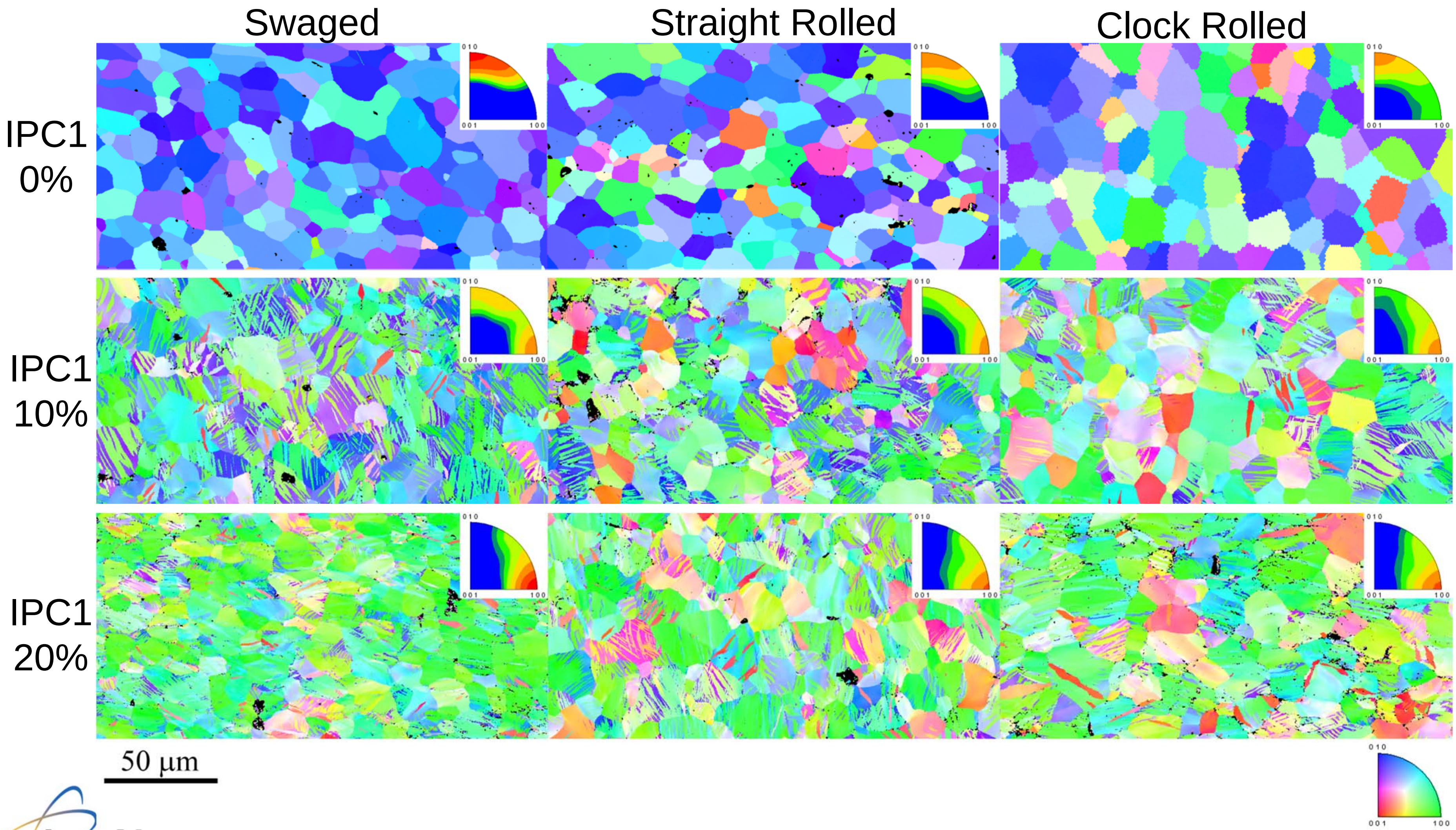
$\{130\}\langle 3\bar{1}0\rangle$	$'\{172\}'\langle 3\bar{1}2\rangle$	$\{112\}'\langle 3\bar{7}2\rangle'$	$'\{176\}'\langle 51\bar{2}\rangle$
2 Twin Variants	4 Twin Variants	4 Twin Variants	4 Twin Variants

α-Uranium Deformation

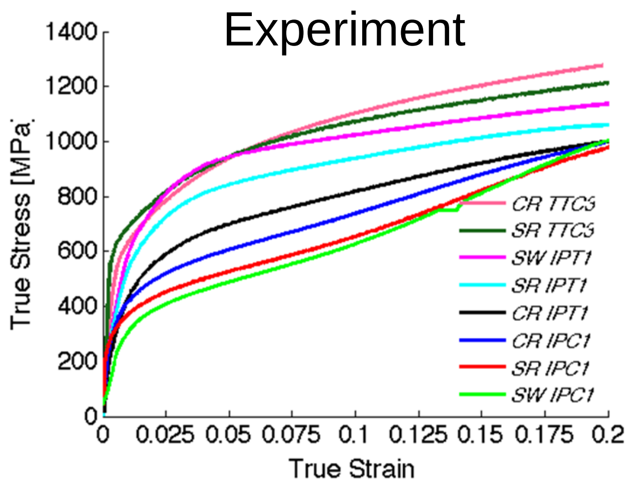


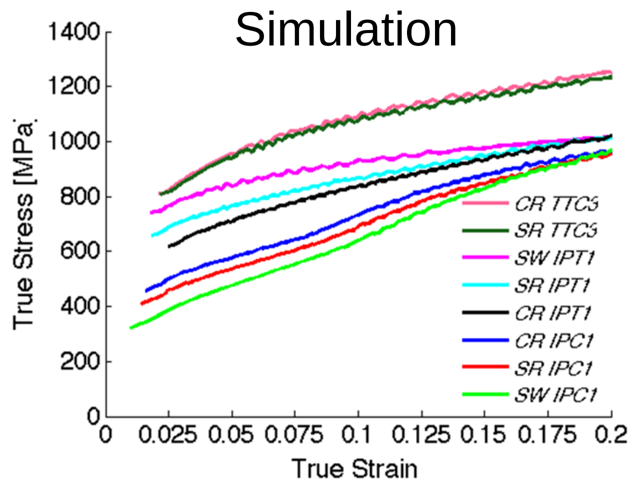


Uranium: Microstructure Change

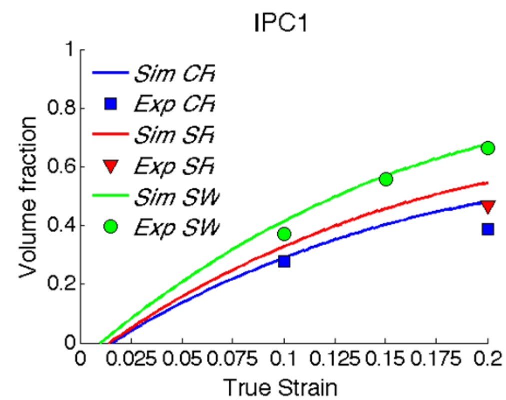


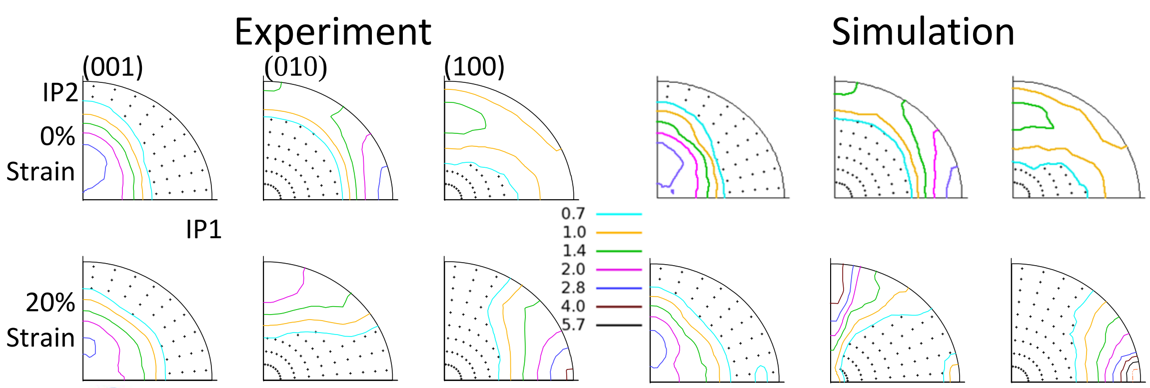
Constitutive Law Development





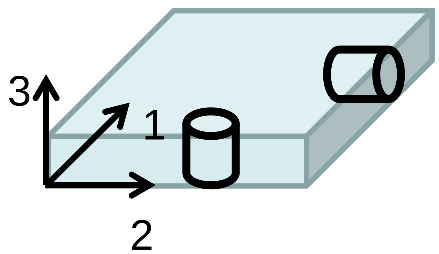
VPSC model predicts uranium anisotropic deformation behavior

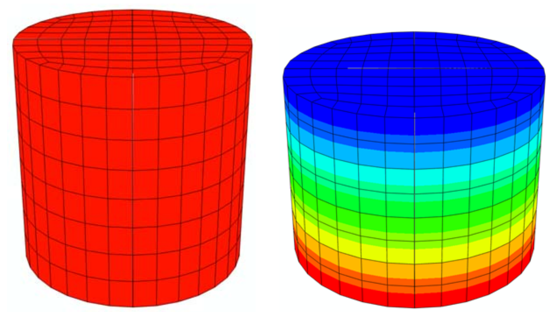


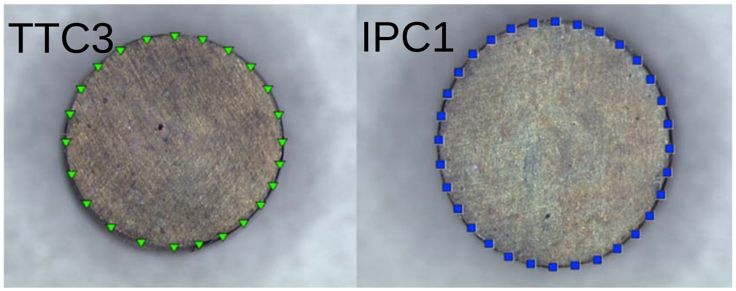


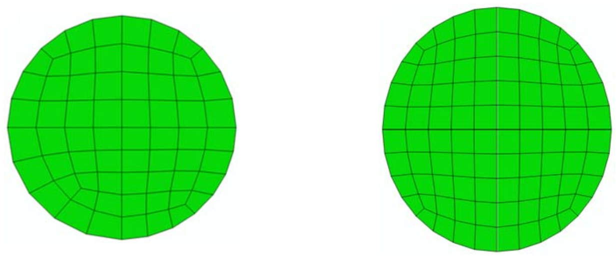
Pole figure texture plots for CR-IPC1 deformation showing that VPSC predicts microstructure evolution

Model Validation: Compression Geometry

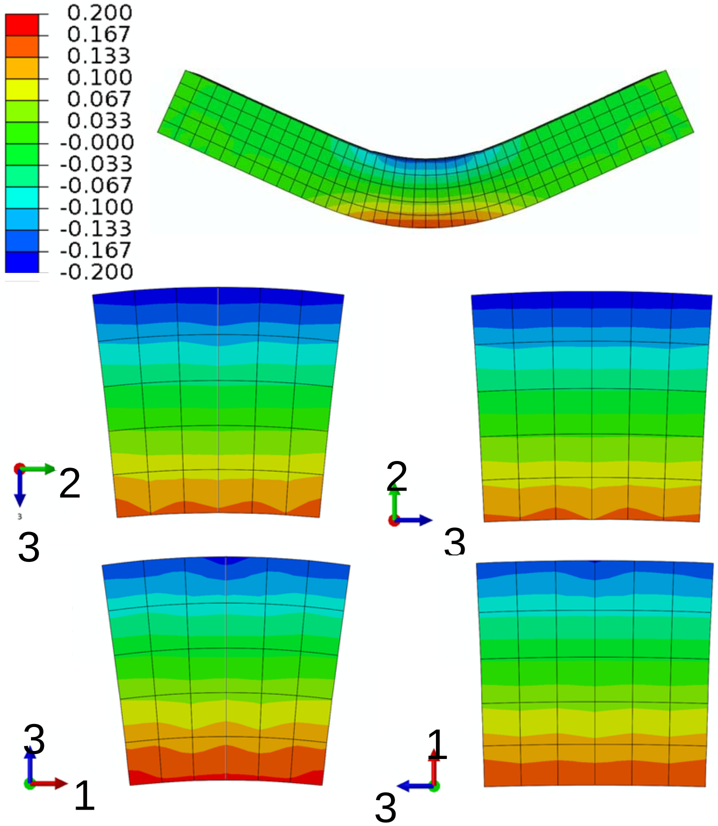


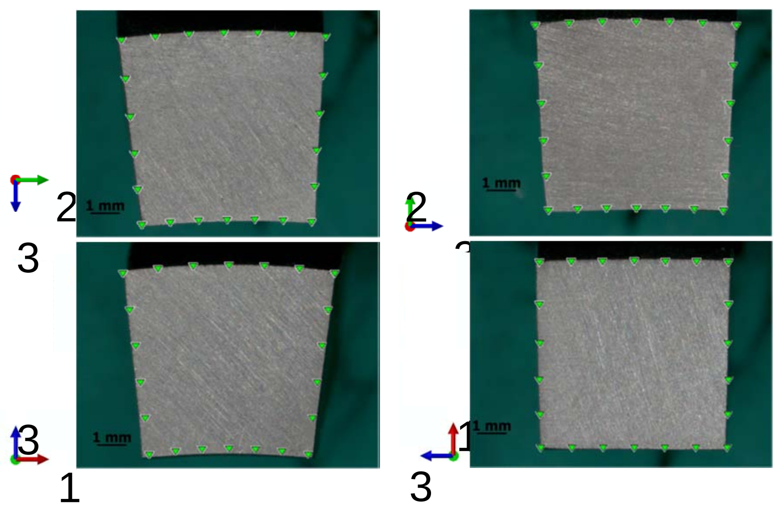






Model Validation: Bent Beams





α-Uranium Fracture

Summary: EBSD of Uranium Alloys

- We continue to improve in our ability to get good EBSD results for uranium alloys
- U-10Mo
  - Characterizing microstructural evolution for a new process
- U-6Nb
  - Corroboration of shape memory mechanical model
- Unalloyed Uranium
  - Development of uranium constitutive deformation law
  - Processing effects on microstructure